# NASA Advances in Colorless and Low Dielectric Polyimide Thin Film Technology

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Aircraft Design, Testing and Performance Chemistry and Materials (General) Composite Materials Metals and Metallic Materials Nonmetallic Materials
Mechanical Engineering
Solid-State Physics

# Aircraft Design, Testing and Performance

Includes all stages of design of aircraft and aircraft structures and systems. Also includes aircraft testing, performance, and evaluation, and aircraft and flight simulation technology.

## 19990005657

Microstructural characterization of semi-interpenetrating polymer networks by positron lifetime spectroscopy

Singh, Jag J.; Pater, Ruth H.; Eftekhari, Abe; Nuclear Instruments & Methods in Physics Research, Section B: Beam Interactions with Materials and Atoms; Jan, 1998; ISSN 0168-583X; Volume 134, no. 1, pp. 113-120; In English; Copyright; Avail: Issuing Activity

Thermoset and thermoplastic polyimides have complementary physical/mechanical properties. Whereas thermoset polyimides are brittle and generally easier to process, thermoplastic polyimides are tough but harder to process. It is expected that a combination of these two types of polyimides may help produce polymers more suitable for aerospace applications. Semi-Interpenetrating Polymer Networks (S-IPNs) of thermoset LaRC (trademark)-RP46 and thermoplastic LaRC (trademark)-IA polyimides were prepared in weight percent ratios ranging from 100:0 to 0:100. Positron lifetime measurements were made in these samples to correlate their free volume features with physical/mechanical properties. As expected, positronium atoms are not formed in these samples. The second life time component has been used to infer the positron trap dimensions. The 'free volume' goes through a minimum at about 50:50 ratio, suggesting that S-IPN samples are not merely solid solutions of the two polymers. These data and related structural properties of the S-IPN samples have been discussed in this paper.

Author (EI)

Polymerization; Thermosetting Resins; Polyimides; Thermoplastic Resins

**TOP** 

# **Chemistry and Materials (General)**

Includes general research topics related to the composition, properties, structure, and use of chemical compounds and materials as they relate to aircraft, launch vehicles, and spacecraft.

19890064843 NASA Langley Research Center, Hampton, VA, USA

X-ray diffraction studies of model compounds of thermoplastic polyimides

Wakelyn, N. T., NASA Langley Research Center, USA; Pratt, J. R., Planning Research Corp., USA; Journal of Polymer Science: Polymer Chemistry Edition; Jan 1, 1989; ISSN 0360-6376; 27, pp. 2833-283; In English; Copyright; Avail: Issuing Activity

Several model compounds which represent aspects of the chemical structure of the Langley Research Center thermoplastic (LARC-TPI) have been synthesized for comparison studies using wide angle X-ray scattering (WAXS) as the discriminatory tool. The model compounds included N-phenylphthalimide, 3,3',4,4'-benzophenonetetracarboxylic diphthalimide, and N,N'-diphenyl-3,3',4,4'-benzophenonetetracarboxylic diphthalimide. X-ray diffraction studies of appropriate model compounds can yield information to aid in the understanding of polymeric crystallographic structure. Crystallinity and physical property changes can thus be induced into materials having complicated structures by chemical and thermal processes.

Polyimides; Polymer Matrix Composites; Thermoplastic Resins; X Ray Diffraction

**TOP** 

# Composite Materials

Includes physical, chemical, and mechanical properties of laminates and other composite materials.

19820023570 NASA Langley Research Center, Hampton, VA, USA

Positron lifetime studies in thermoplastic polyimide test specimens

Singh, J. J., NASA Langley Research Center, USA; Stclair, T. L., NASA Langley Research Center, USA; Holt, W. H., NASA Langley Research Center, USA; Mock, W., Jr., NASA Langley Research Center, USA; Jul 1, 1982; 14p; In English

Report No.(s): NASA-TM-84532; NAS 1.15:84532; Avail: CASI; A03, Hardcopy; A01, Microfiche; Original contains color illustrations
Positron lifetime measurements were made in two thermoplastic polyimide materials recently developed at Langley. The long component lifetime values in polyimidesulfone samples are 847 + or - 81 Ps (dry) and 764 + or - 91 Ps (saturated). The corresponding values in LARC thermoplastic imides are 1080 + or - 139 Ps (dry) and 711 + or - 96 Ps (saturated). Clearly, the presence of moisture has greater effect on positron lifetime in LARC thermoplastic imides than in the case of polyimidesulfones. This result is consistent with the photomicrographic observations made on frozen water saturated specimens of these materials.

Life (Durability); Moisture Content; Polyimides; Positrons; Thermoplastic Resins

Effect of annealing history on free volume in thermoplastics

Singh, J. J., NASA Langley Research Center, USA; St.clair, T. L., NASA Langley Research Center, USA; Jul 1, 1986; 11p; In English Contract(s)/Grant(s): RTOP 505-63-91-01

Report No.(s): NASA-TM-87734; NAS 1.15:87734; Avail: CASI; A03, Hardcopy; A01, Microfiche

Two different types of thermoplastic glassy polymers have been investigated for the effects of thermal annealing on their free volumes. It has been observed that free volumes in glassy polymers decrease asymptotically to a steady level after about four thermal anneals lasting for 24 hours at a temperature about 50 C below their glass transition temperatures. These results indicate that composites incorporating properly annealed thermoplastic matrices may not experience any additional internal stresses due to subsequent thermal excursions experienced while in service. CASI

Annealing; Diffusion; Glass; Polyimides; Polymer Matrix Composites; Sorption; Thermoplastic Resins

**TOP** 

19880004763 Acurex Corp., Aerotherm Div., Mountain View, CA, USA

Development of an impact- and solvent-resistant thermoplastic composite matrix, phase 4

Delano, C. B., Acurex Corp., USA; Sep 1, 1987; 118p; In English

Contract(s)/Grant(s): NAS1-16808; RTOP 534-06-23-02

Report No.(s): NASA-CR-178413; NAS 1.26:178413; Avail: CASI; A06, Hardcopy; A02, Microfiche

Polyimides from BTDA with m-phenylenediamine and three aliphatic diamines were prepared in cresol and characterized. Characterization tests included compression strength and modulus, stressed solvent resistance, and melt-flow tests. Efforts to reduce the molecular weights of these polymers by either stoichiometric imbalance or phthalic anhydride end capping produced opacity in the polymer moldings when the stoichiometry was less than 99 percent. Use of 2,4-diaminotoluene in place of the m-phenylenediamine allowed clear polymer moldings to be obtained at all stoichiometries by end capping or stoichiometric imbalance. After melt-flow/molecular-weight studies, carbon fabric composites were prepared from three polyimide compositions containing BTDA, 2,4-diaminotoluene and two aliphatic diamines. Flexural strengths of two of the resins were in excess of 689 MPa (100 ksi) at both room temperature and 93 C. The polyimide from BTDA was selected for scale-up and neat resin characterization tests. The Tg of this polymer was 233 C.

Composite Materials; Impact Resistance; Matrix Materials; Polyimides; Thermoplastic Resins

**TOP** 

19880004769 NASA Langley Research Center, Hampton, VA, USA

An investigation of physical properties of thermoplastic polyimides

Singh, Jag J., NASA Langley Research Center, USA; Stoakley, Diane M., NASA Langley Research Center, USA; Dec 1, 1987; 13p; In English Contract(s)/Grant(s): RTOP 505-63-01-01

Report No.(s): NASA-TM-100530; NAS 1.15:100530; Avail: CASI; A03, Hardcopy; A01, Microfiche

Thermoplastic polyimides are a class of promising high temperature polymers for aerospace applications. NASA-developed LARC-TPI is a prominent member of this family of polymers. Its physical characteristics have been measured as a function of its curing schedule. The results and their possible interpretations are discussed.

Mechanical Properties; Polyimides; Thermoplastic Resins

**TOP** 

## 19880025914

An evaluation of a high temperature thermoplastic polyimide composite

Hartness, J. Timothy, Dayton, University, USA; Jan 1, 1987; 15p; In English; Advanced materials technology '87, Apr. 6-9, 1987, Anaheim, CA, USA; See also A88-13126; Copyright; Avail: Issuing Activity

LARC-TPI is a unique linear aromatic polyimide developed for use as a matrix in graphite-reinforced composites by NASA-Langley. A molding power version of this formulation has been found to exhibit considerable flow by comparison with film or imidized forms, together with a low melt viscosity that could be taken advantage of in prepreg products. Composites fábricated from five different lots of LARC-TPI powders that exhibited significant differences in melt viscosity and residual volatiles are found to possess considerable mechanical property differences.

Carbon Fiber Reinforced Plastics; Polyimides; Refractory Materials; Thermoplastic Resins

**TOP** 

19910002496 Lockheed Engineering and Sciences Co., Hampton, VA, USA

Thermoplastic polyimide NEW-TPI (trademark)

Hou, Tan-Hung, Lockheed Engineering and Sciences Co., USA; Reddy, Rakasi M., Old Dominion Univ., USA; Oct 1, 1990; 40p; In English Contract(s)/Grant(s): NAS1-19000; NAG1-569; RTOP 505-63-50-01 Report No.(s): NASA-CR-187445; NAS 1.26:187445; Avail: CASI; A03, Hardcopy; A01, Microfiche

Thermal and rheological properties of a commercial thermoplastic polyimide, NEW-TPI (trademark), were characterized. The as-received material possesses initially a transient crystallite form with a bimodal distribution in peak melting temperatures. After the melting of the initial crystallite structure, the sample can be recrystallized by various thermal treatments. A bimodal or single modal melting peak distribution is formed for annealing temperatures below or above 360 C, respectively. The recrystallized crystallinities are all transient in nature. The polymers are unable to be recrystallized after being subjected to elevated temperature annealing above 450 C. The recrystallization mechanism was postulated, and a simple kinetics model was found to describe the behavior rather satisfactory under the conditions of prolonged thermal annealing. Rheological measurements made in the linear viscoelastic range support the evidence observed in the thermal analysis. Furthermore, the measurements sustain the manufacturer's recommended processing window of 400 to 420 C for this material.

Crystallites; Polyimides; Recrystallization; Thermoplastic Resins; Thermoplasticity

Interfacial characteristics of high performance carbon fiber/thermoplastic composites with polyimide coupling agents

Whang, Wha-Tzong, National Chiao Tung University, USA; Liu, Wen-Liang, Industrial Technology Research Institute, USA; SAMPE Quarterly; Oct 1, 1990; ISSN 0036-0821; 22, pp. 3-9; In English; Copyright; Avail: Issuing Activity

Being reactive in their precursor state and thermally stable, polyimides were used as coupling agents for high performance thermoplastics matrices/carbon fiber composites. After imidization, polyimides were chemically bonded to carbon fiber through the reaction of carboxylic acid groups in polyimide (PI) precursors with the polar groups on carbon fiber. The adhesion at the polyimide coupling materials-thermoplastic matrix interface was significantly improved by molecular blending of PI coupling agents with thermoplastics due to the polymerization of two monomers of the polyimide precursors in the solution of high temperature thermoplastic matrix. The increment in adhesion when compared with pure ODA/BTDA (4,4'-oxydianiline benzophenone tetracarboxylic acid dianhydride) PI coupling agent was greater than 150 percent for the molecular blending of ODA/BTDA polyimide/PES (polyethersulfone) system.

Carbon Fiber Reinforced Plastics; Interface Stability; Polyimides; Resin Matrix Composites; Thermoplastic Resins

**TOP** 

# 19910043158 Lockheed Engineering and Sciences Co., Hampton, VA, USA

Characterization of thermoplastic polyimide NEW-TPI

Hou, T. H., Lockheed Engineering and Sciences Co., USA; Reddy, R. M., Old Dominion University, USA; SAMPE Quarterly; Jan 1, 1991; ISSN 0036-0821; 22, pp. 38-46; In English

Contract(s)/Grant(s): NAS1-1900; NAG1-569; Copyright; Avail: Issuing Activity

Thermal and rheological properties of a commercial thermoplastic polyimide, NEW-TPI, were characterized. The as-received material possesses initially a transient crystallite form with a bimodal distribution in peak melting temperatures. After the meltings of the initial crystallite structures, the sample can be recrystallized by various thermal treatments. A bimodal or single-modal melting peak distribution is formed for annealing temperatures below or above 360 C, respectively. The recrystallized crystallinities are all transient in nature. The polymers are unable to be recrystallized after being subjected to elevated temperature annealing above 450 C. The recrystallization mechanism was postulated, and a simple kinetics model was found to describe the behavior satisfactorily under conditions of prolonged thermal annealing.

Annealing; Polyimides; Recrystallization; Thermoplastic Resins

TOP

# 19930004228 NASA Langley Research Center, Hampton, VA, USA

A tough high performance composite matrix

Pater, Ruth H., inventor, NASA Langley Research Center, USA; Johnston, Norman J., inventor, NASA Langley Research Center, USA; Oct 27, 1992; 13p; In English; See also N90-26881

Patent Info.: Filed 31 Oct. 1989; NASA-CASE-LAR-14338-1; US-PATENT-5,159,029; US-PATENT-APPL-SN-429514; Avail: US Patent and Trademark Office, Unavail. Microfiche

This invention is a semi-interpenetrating polymer network which includes a high performance thermosetting polyimide having a nadic end group acting as a crosslinking site and a high performance linear thermoplastic polyimide. An improved high temperature matrix resin is provided which is capable of performing in the 200 to 300 C range. This resin has significantly improved toughness and microcracking resistance, excellent processability, mechanical performance and moisture and solvent resistances.

Official Gazette of the U.S. Patent and Trademark Office

Crosslinking; Microcracks; Polyimides; Temperature Effects; Thermoplastic Resins; Thermosetting Resins

<u>TOP</u>

## 19930069410

Melt processible polyimides for high performance composite applications

Wedgewood, Alan R., Du Pont de Nemours & Co., USA; In: International SAMPE Technical Conference, 24th and International SAMPE Metals and Metals Processing Conference, 3rd, Toronto, Canada, Oct. 20-22, 1992, Proceedings. Vol. 24 (A93-53376 23-23); 1992, pp. T385-T398.; In English; Copyright; Avail: Issuing Activity

The Avimid high temperature polyimide thermoplastic family's K3A and K3B systems retain the excellent strength and environmental degradation resistance of K3, while enhancing damage tolerance and processibility; processing may be in either drapeable wet prepreg form for conventional layup and autoclave techniques, or in fully polymerized dry product form for tapes, melt-impregnated towpregs, powder prepregs, and injection-moldable compounds. The great processing versatility of these resins is explored.

High Temperature Environments; Mechanical Properties; Polyimides; Stretch Forming; Thermoplastic Resins; Welding

TOP

# 19940030866 NASA Langley Research Center, Hampton, VA, USA

Tough high performance composite matrix

Pater, Ruth H., inventor, NASA Langley Research Center, USA; Johnston, Norman J., inventor, NASA Langley Research Center, USA; Jul 19, 1994; 11p; In English; Division of US-Patent-Appl-SN-429514, filed 31 Oct. 1989

Patent Info.: Filed 27 Oct. 1992; NASA-CASE-LAR-14239-2; US-PATENT-5,331,063; US-PATENT-APPL-SN-968082; US-PATENT-APPL-SN-429514; Avail: US Patent and Trademark Office

This invention is a semi-interpentrating polymer network which includes a high performance thermosetting polyimide having a nadic end group acting as a crosslinking site and a high performance linear thermoplastic polyimide. Provided is an improved high temperature matrix resin which is capable of performing in the 200 to 300 C range. This resin has significantly improved toughness and microcracking resistance, excellent processability, mechanical performance, and moisture and solvent resistances.

Official Gazette of the U.S. Patent and Trademark Office

Matrix Materials; Polyimide Resins; Polyimides; Refractory Materials; Thermoplastic Resins; Thermosetting Resins

Semi I.P.Ns with a nadimide end-capped benzhydrol imide and fully cyclised polyimides: Thermomechanical and fracture toughness

Durand, Vincent, CMEOTA, France; Senneron, Michel, CMEOTA, France; Pascal, Thierry, CMEOTA, France; Sillion, Bernard, CMEOTA, France; In: Polymides and other high-temperature polymers; Proceedings of the European Technical Symposium on Polymides and High-Temperature Polymers (STEPI 2), 2nd, Montpellier, France, June 4-7, 1991. A95; 1991, pp. 341-346; In English; See also A95-90762; Copyright; Avail: Issuing Activity

For the last few years there has been an expanding demand for high performance polymer matrix composites which can stand a 300 C continuous service temperature. Among the investigated polymeric products, the American P.M.R. (Polymerization of Monomeric Reactants), and particularly the P.M.R 15 nadimide resin, has now reached an industrial development. In 1987 the CEMOTA has developed a new thermosetting nadimide terminated polyimide resin, called IP 960, which has advantages over P.M.R 15 due to its fully imidized structure: chemical stability, low toxicity before cure and suppression of condensation reaction during cure. However, P.M.R 15 and IP 960 are thermosetting resins and lead to crosslinked matrix with low fracture energy. Our approach was to improve the toughness of the IP 960 crosslinked network by using the semi IPN (Interpenetrating Polymer Network) concept. This implied the combination of the crosslinked polynadimide network with a linear thermoplastic polymer. Author (revised by Herner)

Crosslinking; Fracture Strength; High Polymers; Polyimides; Polymer Blends; Polymer Matrix Composites; Polymerization; Thermodynamic Properties; Thermoplastic Resins

**TOP** 

## 19980131058

## The thermal stability of composite based on thermoplastic polyimide containing diphenyl ether unit (POI)

Zhou, Jiang, Chinese Academy of Sciences, Changchun Inst. of Applied Chemistry, China; He, Tianbai, Chinese Academy of Sciences, Changchun Inst. of Applied Chemistry, China; Zhang, Jin, Chinese Academy of Sciences, Changchun Inst. of Applied Chemistry, China; Ding, Mengxian, Chinese Academy of Sciences, Changchun Inst. of Applied Chemistry, China; Journal of Materials Science Letters; May 15, 1996; ISSN 0261-8028; Volume 15,, no. 10, pp. 916, 917; In English; Copyright; Avail: Aeroplus Dispatch

Results of an experimental evaluation of the performance and thermal stability of a composite based on amorphous thermoplastic polyimide containing a diphenyl ether unit (POI) are reported. In the study, the dynamic mechanical analysis (DMA) and thermogravimetry approaches were used to determine the temperature resistance of the material and to obtain information on the kinetics of the thermal decomposition. Two stages in the thermal degradation of the material are identified; experimentally obtained DMA diagrams and thermogravimetric curves are presented.

Thermal Stability; Thermoplastic Resins; Polyimides; Composite Materials

**TOP** 

## 19980160429

# Thermal aging in a carbon fiber reinforced thermoplastic polyimide

Rodeffer, Č. D., Clemson Univ., USA; Ogale, A. A., Clemson Univ., USA; 1994, pp. 1055-1061; In English; Copyright; Avail: Aeroplus Dispatch The present study investigates the effects of thermal aging upon the dynamic mechanical and tensile creep properties of carbon fiber reinforced AVIMID(R) K3B composites. Results from torsional dynamic mechanical analysis (1-2 shear) and transverse tensile creep compliance are presented, as well as shift rates which are useful in predicting the future aging response.

Carbon Fiber Reinforced Plastics; Thermoplastic Resins; Polyimides; Aging (Materials); Temperature Effects; Mechanical Properties

**TOP** 

## 19980191546

## Interlaminar fracture of carbon-thermoplastic polymide composites

Zhou, Jiang, Chinese Academy of Sciences, Changchun Inst. of Applied Chemistry, China; He, Tianbai, Chinese Academy of Sciences, Changchun Inst. of Applied Chemistry, China; Zhang, Jin, Chinese Academy of Sciences, Changchun Inst. of Applied Chemistry, China; Ding, Mengxian, Chinese Academy of Sciences, Changchun Inst. of Applied Chemistry, China; Journal of Materials Science; 1994; ISSN 0022-2461; Volume 29,, no. 11, pp. 2916-2920; In English; Copyright; Avail: Aeroplus Dispatch

Mode I interlaminar fracture of a novel amorphous thermoplastic polyimide reinforced with unidirectional carbon fiber has been studied experimentally using double cantilever beam specimens and SEM. Three kinds of composites were manufactured from different monomeric reactant solutions which were prepared by using different alcohol solvents. The values of fracture toughness of these three composites were measured to construct the crack growth resistance curves (R curves). The contributions from various failure processes to the total fracture toughness were separated, and approximate calculations of these contributions were conducted based on several simplifying assumptions and some data obtained from the fracture surfaces. Though fiber peeling and fiber breakage are observed, interlaminar fracture in the composites studied is primarily controlled by fracture and deformation in the matrix. It is found that the measured fracture toughnesses of the composites differ from each other not only in the propagation values but also in the initial values. A possible reason for this may be variations of matrix ductility in the three composites.

Author (revised by AIAA)

Interlaminar Stress; Carbon Fibers; Polyimides; Fracture Mechanics; Polymer Matrix Composites; Thermoplastic Resins

**TOP** 

# 19980235568 Maverick Corp., Cincinnati, OH USA

# Low-Cost Production of Composite Bushings for Jet Engine Applications Final Report

Gray, Robert A., Maverick Corp., USA; Aug. 1998; 20p; In English Contract(s)/Grant(s): NAS3-27714; RTOP 523-21-13

Report No.(s): NASA/CR-1998-208515; NAs 1.26:208515; E-11293; No Copyright; Avail: CASI; A03, Hardcopy; A01, Microfiche

The objectives of this research program were to reduce the manufacturing costs of variable stator vane bushings by 1) eliminating the expensive carbon fiber braiding operation, 2) replacing the batch mode impregnation, B-stage, and cutting operations with a continuous process, and 3) reducing the molding cycle and machining operations with injection molding to achieve near-net shapes. Braided bushings were successfully fabricated with both AMB-17XLD and AMB-TPD resin systems. The composite bushings achieved high glass transition temperature after post-cure (+300 C) and comparable weight loss to the PNM-15 bushings. ANM-17XLD bushings made with "batch-mode" molding compound (at 0.5 in. fiber length) achieved a +300 lb-force flange break strength which was superior to the continuous braided-fiber reinforced bushing. The non-MDA resin technology developed in this contract appears attractive for bushing applications that do not exceed a 300 C use temperature. Two thermoplastic polyimide resins were synthesized in order to generate injection molding compound powders. Excellent processing results were obtained at injection temperatures in excess of 300 C. Micro-tensile specimens were produced from each resin type and the Tg measurements (by TMA) for these samples were equivalent to AURUM(R). Thermal Gravimetric Analysis (TGA) conducted at 10 C/min showed that the non-MDA AMB-type polyimide thermoplastics had comparable weight loss to PMR-15 up to 500 C.

Jet Engines; Cost Reduction; Fiber Composites; Injection Molding; Machining; Manufacturing; Polyimides; Thermoplastic Resins;

# **Metals and Metallic Materials**

Includes physical, chemical, and mechanical properties of metals and metallic materials; and metallurgy.

#### 19960029791

Thermal stability of composite based on thermoplastic polyimide containing diphenyl ether unit (POI)

Zhou, Jiang, Chinese Acad of Sciences, China; He, Tianbal; Zhang, Jin; Ding, Mengxian; Journal of Materials Science Letters; May 15 1996; ISSN 0261-8028; 15, 10, pp. 916-917; In English; Copyright; Avail: Issuing Activity

A composite material was developed based on an amorphous thermoplastic polyimide containing diphenyl ether unit. In order to evaluate its performance and thermal stability, dynamic mechanical analysis and thermogravimetric analysis were used to gain an insight into the temperature resistance of the material and to obtain information on the kinetics of the thermal decomposition.

Author (FI)

Composite Materials; Diphenyl Compounds; Polyimides; Temperature Effects; Thermal Stability; Thermoplastic Resins

**TOP** 

# **Nonmetallic Materials**

Includes physical, chemical, and mechanical properties of plastics, elastomers, lubricants, polymers, textiles, adhesives, and ceramic materials.

## 19810009673 NASA Langley Research Center, Hampton, VA, USA

Recent developments in polyimide adhesives at NASA-Langley Research Center

St.clair, T. L., NASA Langley Research Center, USA; Jan 1, 1981; 14p; In English Contract(s)/Grant(s): RTOP 505-33-33-02

Report No.(s): NASA-TM-81936; Avail: CASI; A03, Hardcopy; A01, Microfiche

Adhesive development is directed towards elevated temperature applications (200-300 C). Because of thermal stability considerations, the most attractive adhesives for this temperature range are linear and addition polyimides. The linear polymide adhesive research encompassed basic structure-property relationships, solvent studies and formulations to meet various requirements. The most recent research in linear polyimide systems was in the development of thermoplastic systems in an effort to eliminate the undesirable evolution of water classically associated with the cure going through an amide-acid intermediate step in the cure process. Addition polyimide adhesive research was undertaken in order to avoid water evolution during cure. Basic structure-property relationships for these materials led to an adhesive which was used extensively for high temperature adhesive needs. Since addition systems are of a highly crosslinked nature, they are not as resistant to impact as their linear counterparts. In order to overcome this problem, research was done in the area of elastomer-toughening these polymers. S.F.

Adhesives; Crosslinking; Polyimides; Shear Strength; Thermal Stability; Thermoplastic Resins

**TOP** 

## 19810016677 NASA Langley Research Center, Hampton, VA, USA

Flow properties of a series of experimental thermoplastic polymides

Burks, H. D., NASA Langley Research Center, USA; Nelson, J. B., NASA Langley Research Center, USA; Price, H. L., NASA Langley Research Center, USA; Apr 1, 1981; 29p; In English; 52nd; Soc. of Rheology Ann. Meeting, 23-25 Feb. 1981, Williamsburg, VA, USA Contract(s)/Grant(s): RTOP 505-33-33-02

Report No.(s): NASA-TM-83115; Avail: CASI; A03, Hardcopy; A01, Microfiche

The softening temperature to degradation temperature range of the polymers was about 440 to 650 K. All of the polymers retained small amounts of solvent as indicated by an increase in T(sub g) as the polymers were dried. The flow properties showed that all three polymers had very high apparent viscosities and would require high pressures and/or high temperatures and/or long times to obtain adequate flow in prepregging and molding. Although none was intended for such application, two of the polymers were combined with carbon fibers by solution prepregging. The prepregs were molded into laminates at temperatures and times, the selection of which was guided by the results from the flow measurements. These laminates had room temperature short beam shear strength similar to that of carbon fiber laminates with a thermosetting polyimide matrix. However, the strength had considerable scatter, and given the difficult processing, these polymides probably would not be suitable for continuous fiber composites. E.D.K.

Carbon Fiber Reinforced Plastics; Flow Characteristics; Polyimides; Rheometers; Thermoplastic Resins

**TOP** 

# 19810018740 NASA Langley Research Center, Hampton, VA, USA

A review of high-temperature adhesives

St.clair, A. K., NASA Langley Research Center, USA; St.clair, T. L., NASA Langley Research Center, USA; Jul 1, 1981; 22p; In English Contract(s)/Grant(s): RTOP 505-33-33-02

Report No.(s): NASA-TM-83141; Avail: CASI; A03, Hardcopy; A01, Microfiche

The development of high temperature adhesives and polyphenylquinoxalines (PPQ) is reported. Thermoplastic polyimides and linear PPQ adhesive are shown to have potential for bonding both metals and composite structures. A nadic terminated addition polyimide adhesive, LARC-13, and an acetylene terminated phenylquinoxaline (ATPQ) were developed. Both of the addition type adhesives are shown to be more readily processable than linear materials but less thermooxidatively stable and more brittle. It is found that the addition type adhesives are able to perform, at elevated temperatures up to 595 C where linear systems fail thermoplastically.

F A K

Adhesives; High Temperature; Linearity; Polyimides; Polyquinoxalines; Thermoplastic Resins; Tolerances (Mechanics)

<u>TOP</u>

The development of aerospace polyimide adhesives

St.clair, A. K., NASA Langley Research Center, USA; St.clair, T. L., NASA Langley Research Center, USA; Jan 1, 1983; 28p; In English; 1st; Tech. Conf. on Polyimides, Nov. 1982, Ellenville, NY, USA

Contract(s)/Grant(s): RTOP 505-33-33-09

Report No.(s): NASA-TM-84587; NAS 1.15:84587; Avail: CASI; A03, Hardcopy; A01, Microfiche

Few materials are available which can be used as aerospace adhesives at temperatures in the range of 300 C. The Materials Division at NASA-Langley Research Center developed several high temperature polyimide adhesives to fulfill the stringent needs of current aerospace programs. These adhesives are the result of a decade of basic research studies on the structure property relationships of both linear and addition aromatic polyimides. The development of both in house and commercially available polyimides is reviewed with regards to their potential for use as aerospace adhesives.

Adhesives; Polyimides; Polymerization; Spacecraft Construction Materials; Thermoplastic Resins

**TOP** 

## 19830025770 NASA Langley Research Center, Hampton, VA, USA

Solvent resistant thermoplastic aromatic poly(imidesulfone) and process for preparing same

St.clair, T. L., NASA Langley Research Center, USA; Yamaki, D. A., inventors, NASA Langley Research Center, USA; Aug 9, 1983; 4p; In English; See also N83-13259

Patent Info.: Filed 11 Aug. 1982; NASA-CASE-LAR-12858-1; US-PATENT-4,398,021; US-PATENT-APPL-SN-407240; Avail: US Patent and Trademark Office, Unavail. Microfiche

A process for preparing a thermoplastic poly(imidesulfone) is disclosed. This resulting material has thermoplastic properties which are generally associated with polysulfones but not polysulfones. This system is processable in the 250 to 350 C range for molding, adhesive and laminating applications. This unique thermoplastic poly(imidesulfone) is obtained by incorporating an aromatic sulfone moiety into the backbone of an aromatic linear polyimide by dissolving a quantity of a 3,3',4,4'-benzophenonetetracarboxylic dianhydride (BTDA) in a solution of 3,3'-diaminodiphenylsulfone and bis(2-methoxyethyl)ether, precipitating the reactant product in water, filtering and drying the recovered poly(amide-acid sulfone) and converting it to the poly(imidesulfone) by heating.

Official Gazette of the U.S. Patent and Trademark Office

Polyimides; Polymerization; Sulfones; Synthesis (Chemistry); Thermoplastic Resins

TOP

## 19850004653 NASA Langley Research Center, Hampton, VA, USA

Thermoplastic/melt-processable polyimides

St.clair, T. L., NASA Langley Research Center, USA; Burks, H. D., NASA Langley Research Center, USA; Tough Composite Mater.; Dec 1, 1984, pp. p 337-355; In English; See also N85-12941 04-24; A03

Several polyimides were prepared which show promise for aircraft composite applications. This was achieved through a systematic polymer synthesis program where the glass transition temperatures were greatly lowered when compared to the older polyimide systems. Several of the materials were shown to be hot-melt processable and are attractive matrix resin candidates especially in light of their high g(sub lc) values. At least two of these polyimides are available for evaluation and others are on the research horizon.

Molecular Weight; Polyimides; Synthesis (Chemistry); Thermoplastic Resins

**TOP** 

# 19850010825 NASA Langley Research Center, Hampton, VA, USA

Characterization of a thermoplastic polyimidesulfone

Dezern, J. F., NASA Langley Research Center, USA; Young, P. R., NASA Langley Research Center, USA; Feb 1, 1985; 37p; In English Contract(s)/Grant(s): RTOP 505-33-33-02

Report No.(s): NASA-TM-86358; NAS 1.15:86358; Avail: CASI; A03, Hardcopy; A01, Microfiche

The detailed characterization of an experimental thermoplastic polyimidesulfone adhesive based on 3,3 prime-diaminodiphenylsulfone and 3,3 prime,4,4 prime-benzophenone tetracarboxylic dianhydride was studied. Model compounds were also examined. Thermal cyclization of the amide-acid to the imide was studied by a variety of techniques including DSC, TGA, MS, in situ diffuse reflectance-FTIR, and flow mearsurement. Characterizations were continued during the processing of adhesive tapes and the fabrication, bonding, and testing of lap shear specimens. Results provide fundamental insights into the role of cure chemistry, and the effects of residual solvent and volatile produces on processing and performance. These insights and the resulting chemical models should lead to more efficient processing cycles for these and other related thermoplastic adhesive systems.

 ${\bf Adhesive\ Bonding;\ Curing;\ Polyimides;\ Sulfones;\ Thermoplastic\ Resins}$ 

TOP

# 19850039370 NASA Langley Research Center, Hampton, VA, USA

The development of aerospace polyimide adhesives

St. Clair, A. K., NASA Langley Research Center, USA; St. Clair, T. L., NASA Langley Research Center, USA; Jan 1, 1984; 26p; In English; See also A85-21476 08-27; Copyright; Avail: Issuing Activity

Few materials are available which can be used as aerospace adhesives at temperatures in the range of 300 C. The Materials Division at NASA-Langley Research Center developed several high temperature polyimide adhesives to fulfill the stringent needs of current aerospace programs. These adhesives are the result of a decade of basic research studies on the structure property relationships of both linear and addition aromatic polyimides. The development of both in house and commercially available polyimides is reviewed with regards to their potential for use as aerospace adhesives.

AIAA

Adhesives; Polyimides; Polymerization; Spacecraft Construction Materials; Thermoplastic Resins

19850059620 Kentron International, Inc., Hampton, VA, USA

Semi-2-interpenetrating polymer networks of high temperature systems

Hanky, A. O., Kentron International, Inc., USA; St. Clair, T. L., NASA Langley Research Center, USA; SAMPE Journal; Aug 1, 1985; ISSN 0091-1062; 21, pp. 40-45; In English; Copyright; Avail: Issuing Activity

A semi-interpenetrating (semi-IPN) polymer system of the semi-2-IPN type is described in which a polymer of acetylene-terminated imidesulfone (ATPISO2) is cross linked in the presence of polyimidesulfone (PISO2). Six different formulations obtained by mixing of either ATPISO2-1n or ATPISO2-3n with PISO2 in three different proportions were characterized in terms of glass transition temperature, thermooxidative stability, inherent viscosity, and dynamic mechanical properties. Adhesive (lap shear) strength was tested at elevated temperatures on aged samples of adhesive scrim cloth prepared from each resin. Woven graphite (Celion 1000)/polyimide composites were tested for flexural strength, flexural modulus, and shear strength. The network polymers have properties intermediate between those of the component polymers alone, have greatly improved processability over either polyimide, and are able to form good adhesive bonds and composites, making the semi-2-IPN systems superior materials for aerospace structures. AIAA

Adhesive Bonding; Carbon Fiber Reinforced Plastics; Mechanical Properties; Polyimides; Thermoplastic Resins

**TOP** 

19860001872 College of William and Mary, Dept. of Chemistry., Williamsburg, VA, USA

Characterization of the relationship of the cure cycle chemistry to cure cycle processing properties Report, period ending 31 Jul. 1985 Kranbuehl, D. E., College of William and Mary, USA; Aug 23, 1985; 33p; In English Contract(s)/Grant(s): NAG1-237

Report No.(s): NASA-CR-176256; NAS 1.26:176256; Avail: CASI; A03, Hardcopy; A01, Microfiche

Dynamic dielectric analysis (DDA) is used to study curing polymer systems and thermoplastics. Measurements are made over a frequency range of six decades. This wide range of frequencies increases the amount of information which can be obtained. The data is analyzed in terms of the frequency dependence of the complex permittivity epsilon sup \*, specific conductivity sigma (ohm/cm) and the relaxation time tau, parameters which are characteristic of the cure state of the material and independent of the size of the sample.

Curing; Dielectric Properties; Dynamic Tests; Epoxy Resins; Evolution (Liberation); Nondestructive Tests; Polyimides; Polymer Chemistry; Polyphenyls; Solvents; Thermoplastic Resins; Viscosity

**TOP** 

19860017981 NASA Langley Research Center, Hampton, VA, USA

Adhesive evaluation of thin films of LARC-TPI and LARC-TPI with 5 mol % ODA

Progar, D. J., NASA Langley Research Center, USA; Jun 1, 1986; 30p; In English Contract(s)/Grant(s): RTOP 506-43-11

Report No.(s): NASA-TM-87738; NAS 1.15:87738; Avail: CASI; A03, Hardcopy; A01, Microfiche

A commercially available LARC-TPI film and an experimentally prepared film of LARC-TPI with 5 mol % of 4,4'-oxydianiline (ODA), designated as LARC-TPI/ODA in the report, supplied by Mitsui Toatsu Chemicals, Incorporated (MTCI), Japan, were evaluated as thermoplastic adhesive films for bonding Ti-6AI-4V. The LARC-TPI/ODA had been shown by MTCI to possess more flow than thermoplastic LARC-TPI and was, therefore, evaluated and compared to the LARC-TPI. Lap shear strength was used to evaluate the materials as adhesives. They were characterized after fracture by determining the glass transition temperature, Tg. The mode of failure was also reported. Thermal exposure at 204C for 500 and 1000 hrs and a 72-hour water-boil were conducted on lap shear specimens prepared with the two adhesive films. Lap shear tests were conducted at RT, 177C, 204C, and 232C before and after exposures. CASI

Adhesive Bonding; Evaluation; Lap Joints; Polyimides; Shear Strength; Thermoplastic Films; Thin Films

**TOP** 

## 19860028392

AIAA

## Preparation and characterization of thermo-plastic/thermo-setting polyimide blends

Yamamoto, Y.; Satoh, S.; Etoh, S., Mitsubishi Electric Corp., Japan; Jan 1, 1985; 9p; In English; See also A86-13076; Copyright; Avail: Issuing Activity

Thermoplastic/thermosetting polyimide blends have been prepared using soluble polyimide (Pl2080) and N, N-prime-(methylenedi-p-phenylene)-bismaleimide (BMI). The blends were characterized and evaluated to determine their potential as a composite matrix resin. The blends were found to polymerize at temperatures of 180 C or above. High glass transition temperatures were achieved after a prolonged postcure, such as 16 hours at 240 C. Pl2080/BMI ratio influenced both the flow property and thermal stability of the blends, but did not have a significant effect on the glass transition temperature. From the viewpoint of the processability and thermal stability, a PI2080/BMI 35/65 weight-ratio blend was selected for a more detailed evaluation. Carbon and glass fabric composites with glass transition temperatures in excess of 300 C were made. The carbon fabric composite showed 70 percent flexural strength retention at ambient temperature after aging at 240 C for 1000 hours. AIAÀ

Mechanical Properties; Polyimides; Polymer Matrix Composites; Thermoplastic Resins

**TOP** 

19860036811 NASA Langley Research Center, Hampton, VA, USA

Process optimization of a thermoplastic polyimidesulphone

Dezern, J. F., NASA Langley Research Center, USA; Young, P. R., NASA Langley Research Center, USA; International Journal of Adhesion and Adhesives; Oct 1, 1985; ISSN 0143-7496; 5, pp. 183-192; In English; Previously announced in STAR as N85-19135; Copyright; Avail: Issuing Activity The detailed characterization of an experimental thermoplastic polyimidesulfane adhesive based on 3,3 prime-diaminodiphenylsulfone and 3,3 prime,4,4 prime-benzophenone tetracarboxylic dianhydride was studied. Model compounds were also examined. Thermal cyclization of the amide-acid to the imide was studied by a variety of techniques including DSC, TGA, MS, in situ diffuse reflectance-FTIR, and flow measurement. Characterizations were continued during the processing of adhesive tapes and the fabrication, bonding, and testing of lap shear specimens. Results provide fundamental insights into the role of cure chemistry, and the effects of residual solvent and volatile produces on processing and performance. These insights and the resulting chemical models should lead to more efficient processing cycles for these and other related thermoplastic adhesive systems.

Adhesive Bonding; Curing; Polyimides; Sulfones; Thermoplastic Resins

Evaluation of a novel thermoplastic polyimide for bonding titanium

Progar, D. J., NASA Langley Research Center, USA; St. Clair, T. L., NASA Langley Research Center, USA; International Journal of Adhesion and Adhesives; Jan 1, 1986; ISSN 0143-7496; 6, pp. 25-30; In English; Copyright; Avail: Issuing Activity

A novel hot-melt processable polyimide has been synthesized and characterized as an adhesive for a titanium alloy. This system shows potential for applications with service temperatures near 200 C. A bonding cycle was developed from preliminary processing studies. The bonded specimens were tested at room temperature, 177 C and 204 C both before and after aging in air at 204 C for times up to 1000 hours. There was no appreciable change in strength after aging. Bonded specimens were exposed to boiling water continuously for a 72 hour period. These specimens exhibited some loss in strength due to this rather severe exposure.

Adhesive Bonding; Metal-Metal Bonding; Polyimides; Shear Strength; Thermoplastic Resins; Titanium Alloys

**TOP** 

# 19870012620 NASA Langley Research Center, Hampton, VA, USA

Adhesive evaluation of new polyimides

Stclair, Terry L., NASA Langley Research Center, USA; Progar, Donald J., NASA Langley Research Center, USA; Apr 1, 1987; 6p; In English Contract(s)/Grant(s): RTOP 506-43-11-01

Report No.(s): NASA-TM-89140; NAS 1.15:89140; Avail: CASI; A02, Hardcopy; A01, Microfiche

During the past 10 to 15 years, the Materials Division at NASA Langley Research Center (LaRC) has developed several novel high temperature polyimide adhesives for anticipated needs of the aerospace industry. These developments have resulted from fundamental studies of structure-property relationships in polyimides. Recent research at LaRC has involved the synthesis and evaluation of copolyimides which incorporate both flexibilizing bridging groups and meta-linked benzene rings. The purpose was to develop systems based on low cost, readily available monomers. Two of these copolyimides evaluated as adhesives for bonding titanium alloy, Ti(6Al-4V), are identified as LARC-STPI and STPI-LARC-2. Lap shear strength (LSS) measurements were used to determine the strength and durability of the adhesive materials. LSS results are presented for LARC-TPI and LARC-STPI lap shear specimens thermally exposed in air at 232 C for up to 5000 hrs. LARC-TPI was shown to perform better than the copolymer LARC-STPI which exhibited poor thermooxidative performance possibly due to the amines used which would tend to oxidize easier than the benzophenone system in LARC-TPI.

Adhesives; High Temperature; Polyimides; Thermoplastic Resins

**TOP** 

# 19870036427 NASA Langley Research Center, Hampton, VA, USA

Characterization of crystalline LARC-TPI powder

Burks, Harold D., NASA Langley Research Center, USA; Saint Clair, Telly L., NASA Langley Research Center, USA; Hou, Tan-Hung, PRC Kentron, Inc., USA; SAMPE Quarterly; Oct 1, 1986; ISSN 0036-0821; 18, pp. 1-8; In English; Copyright; Avail: Issuing Activity

LARC-TPI, a linear aromatic polyimide, developed by NASA Langley Research Center in the late 1970's had thermoplastic character, but its melt-flow properties were much lower than those of conventional thermoplastics. An imidized version of the LARC-TPI molding powder was submitted to NASA-Langley for evaluation. The initial broad melt of this material occurs in the 270 C range and reorders to higher melting forms, depending on the physical and thermal treatments. Rheological characterization of this polymer shows it to have a very low melt viscosity, which increases as the melted specie transforms to other crystalline forms with higher melting temperatures.

Aromatic Compounds; Polyimides; Rheology; Thermal Stability; Thermoplastic Resins

<u>TOP</u>

## 19870040864

LARC-TPI and new thermoplastic polyimides

Yamaguchi, Akihiro; Ohta, Masahiro, Mitsui Toatsu Chemicals, Inc., Japan; SAMPE Journal; Feb 1, 1987; ISSN 0091-1062; 23, pp. 28-32; In English; Copyright; Avail: Issuing Activity

The LARC-TPI linear thermoplastic polyimide has been developed by NASA for high temperature adhesive applications in aerospace structures in the forms of varnish, films, powders, and prepregs. LARC-TPI improves adhesive processability and lowers glass transition temperature, while retaining mechanical, thermal and electrical properties inherent in the polyimides. It may be used as a structural adhesive for metals, composites, ceramics, and films.

Adhesive Bonding; Aromatic Compounds; Injection Molding; Mechanical Properties; Polyimides; Thermoplastic Resins

**TOP** 

# 19880004092 NASA Langley Research Center, Hampton, VA, USA

Flexibilized copolyimide adhesives

Progar, Donald J., NASA Langley Research Center, USA; St.clair, Terry L., NASA Langley Research Center, USA; Jan 1, 1988; 40p; In English; Submitted for publication

Report No.(s): NASA-TM-89303; NAS 1.15:89303; Avail: CASI; A03, Hardcopy; A01, Microfiche

Two copolyimides, LARC-STPI and STPI-LARC-2, with flexible backbones were processed and characterized as adhesives. The processability and adhesive properties were compared to those of a commercially available form of LARC-TPI. Lap shear specimens were fabricated using adhesive tape prepared from each of the three polymers. Lap shear tests were performed at room temperature, 177 C, and 204 C before and after exposure to water-boil and to thermal aging at 204 C for up to 1000 hours. The three adhesive systems possess exceptional lap shear strengths at room temperature and elevated temperatures both before and after thermal exposure. LARC-STPI, because of its high glass transition temperature provided high lap shear strengths up to 260 C. After water-boil, LARC-TPI exhibited the highest lap shear strengths at room temperature and 177 C, whereas the LARC-STPI retained a higher percentage of its original strength when tested at 204 C. These flexible thermoplastic copolyimides show considerable potential as adhesives based on this study and because of the ease of preparation with low cost, commercially available materials.

Adhesive Bonding; Adhesives; Copolymers; Flexibility; Polyimides; Shear Strength; Thermoplastic Resins

Evaluation of a thermoplastic polyimide (422) for bonding GR/PI composite

Progar, Donald J., NASA Langley Research Center, USA; Apr 1, 1988; 28p; In English

Contract(s)/Grant(s): RTOP 505-63-01-01

Report No.(s): NASA-TM-100584; NAS 1.15:100584; Avail: CASI; A03, Hardcopy; A01, Microfiche

A hot-melt processable copolyimide previously studied and characterized as an adhesive for bonding Ti-6Al-4V was used to bond Celion 6000/LARC-160 composite. Comparisons are made for the two adherend systems. A bonding cycle was determined for the composite bonding and lap shear specimens were prepared which were thermally exposed in a forced-air oven for up to 5000 h at 204 C. The lap shear strengths (LSSs) were determined at RT, 177, and 204 C. After thermal exposure at RT, 177, and 204 C the LSS decreased significantly; however, a slight increase was noted for the 204 C tests. Initially the LSS values are higher for the bonded Ti-6Al-4V than for the bonded composite, however, the LSS decreases dramatically between 5000 and 10,000 h of 204 C thermal exposure. Longer periods of thermal exposure up to 20,000 h results in further decreases in the LSSs. Although the bonded composite retained useful strengths for exposures up to 5000 h, based on the poor results of the bonded Ti-6Al-4V beyond 5000 h, the 422 adhesive bonded composites would most likely also produce poor strengths beyond 5000 h exposure. Adhesive bonded composite lap shear specimens exposed to boiling water for 72 h exhibited greatly reduced strengths at all test temperatures. The percent retained after water boil for each test temperature was essentially the same for both systems.

Adhesive Bonding; Polyimides; Shear Strength; Synthesis (Chemistry); Thermoplastic Resins

**TOP** 

# 19880031613 NASA Langley Research Center, Hampton, VA, USA

High temperature polyimide blends

Burks, Harold D., NASA Langley Research Center, USA; St. Clair, Terry L., NASA Langley Research Center, USA; SAMPE Quarterly; Oct 1, 1987; ISSN 0036-0821; 19, pp. 1-6; In English; Copyright; Avail: Issuing Activity

In a continuing effort to synthesize polymers that are readily processable and that exhibit state-of-the-art characteristics, LARC-TPI (Mitsui) and polyimidesulfone (PISO2) were blended and subjected to a limited melt flow and thermal properties characterization. Mitsui Toatsu of Japan, working under a licensing agreement with NASA, synthesized an imidized version of LARC-TPI molding powder that exhibited an extraordinarily high level of softening when heated to 300 C. Subsequently, a study of the calorimetric and rheological properties was accomplished by NASA-Langley on that material. The thermoplastic polyimidesulfone that possesses processability similar to polysulfones and the solvent resistance of the polyimides was synthesized and characterized at NASA-Langley in the early 1980's.

Polyimides; Polymer Chemistry; Synthesis (Chemistry); Thermal Stability; Thermoplastic Resins

**TOP** 

# 19880036965 NASA Langley Research Center, Hampton, VA, USA

Evaluation of polyimide films as adhesives

Progar, Donald J., NASA Langley Research Center, USA; Journal of Adhesion Science and Technology; Jan 1, 1987; ISSN 0169-4243; 1, 1, 19; 16p; In English; Copyright; Avail: Issuing Activity

A commercially available LARC-TPI film and an experimentally prepared film of LARC-TPI with 5 mol pct of 4,4-prime-oxydianiline (ODA), designated as LARC-TPI/ODA, were evaluated as thermoplastic adhesive films for bonding Ti-6Al-4V. Lap shear strength was used to evaluate the materials as adhesives. They were characterized after fracture by determining the glass transition temperature, T(g). The mode of failure is also reported. Thermal exposure at 240 C for 500 and 1000 h and a 72-h water-boil were conducted on lap shear specimens prepared with the two adhesive films. Lap shear tests were conducted at room temperature, 177 C, 204 C, and 232 C before and after exposures. AIAA

Adhesive Bonding; Glass Transition Temperature; Polyimides; Polymeric Films; Thermoplastic Resins; Thin Films

**TOP** 

# 19890005555 Akron Univ., Dept. of Polymer Engineering., Akron, OH, USA

Polymer powder prepregging: Scoping study Abstract Only

Throne, James L., Akron Univ., USA; Hampton Inst., NASA/American Society for Engineering Edu; Sep 1, 1988, pp. Hampton Inst., NASA; In English; Avail: Issuing Activity

Early on, it was found that NEAT LARC-TPI thermoplastic polyimide powder behaved elastoplastically at pressures to 20 ksi and temperatures to 260 degrees celcius (below MP). At high resin assay, resin powder could be continuously cold-flowed around individual carbon fibers in a metal rolling mill. At low resin assay (2:1, C:TPI), fiber breakage was prohibitive. Thus, although processing of TPI below MP would be quite unique, it appears that the polymer must be melted and flowed to produce low resin assay prepreg. Fiber tow was spread to 75 mm using a venturi slot tunnel. This allowed intimate powder/fiber interaction. Two techniques were examined for getting room temperature powder onto the room temperature fiber surface. Electrostatic powder coating allows the charged powder to cling tenaciously to the fiber, even while heated with a hot air gun to above its melt temperature. A variant of the wet slurry coating process was also explored. The carbon fibers are first wetted with water. Then dry powder is sprinkled onto the wet tow and doctor-rolled between the fibers. The wet structure is then taken onto a heated roll, with hot air guns drying and sinter-melting the powder onto the fiber surfaces. In both cases SEM shows individual fibers coated with powder particles that have melted in place and flowed along the fiber surface via surface tension.

Carbon Fibers; Polyimides; Powder (Particles); Prepregs; Process Control (Industry); Thermoplastic Resins

**TOP** 

# 19890015117 Clemson Univ., Center for Advanced Engineering Fibers., SC, USA

Thermoplastic coating of carbon fibers Topical Annual Report, 1988 - 1989

Edie, D. D., Clemson Univ., USA; Lickfield, G. C., Clemson Univ., USA; Allen, L. E., Clemson Univ., USA; Mccollum, J. R., Clemson Univ., USA; Jan 1, 1989; 136p; In English Contract(s)/Grant(s): NAG1-680

Report No.(s): NASA-CR-185047; NAS 1.26:185047; Avail: CASI; A07, Hardcopy; A02, Microfiche

A continuous powder coating system was developed for coating carbon fiber with LaRC-TPI (Langley Research Center-Thermoplastic Polyimide), a high-temperature thermoplastic polymide invented by NASA-Langley. The coating line developed used a pneumatic fiber spreader to separate the individual fibers. The polymer was applied within a recirculating powder coating chamber then melted using a combination of direct electrical resistance and convective heating to make it adhere to the fiber tow. The tension and speed of the line were controlled with a dancer arm and an electrically driven fiber wind-up and wind-off. The effects of heating during the coating process on the flexibility of the prepreg produced were investigated. The uniformity with which the fiber tow could be coated with polymer also was examined. Composite specimens were fabricated from the prepreg and tested to determine optimum process conditions. The study showed that a very uniform and flexible prepeg with up to 50 percent by volume polymer could be produced with this powder coating system. The coating line minimized powder loss and produced prepeg in lengths of up to 300 m. The fiber spreading was found to have a major effect on the coating uniformity and flexibility. Though test results showed low composite tensile strengths, analysis of fracture surfaces under scanning electron microscope indicated that fiber/matrix adhesion was adequate.

**TOP** 

# 19890041709 NASA Langley Research Center, Hampton, VA, USA

Evaluation of a thermoplastic polyimide (422) for bonding GR/PI composite

Progar, Donald J., NASA Langley Research Center, USA; Journal of Adhesion Science and Technology; Jan 1, 1988; ISSN 0169-4243; 2, 6, 19, pp. 449-461; In English; Previously announced in STAR as N88-230; Copyright; Avail: Issuing Activity

A hot-melt processable copolyimide previously studied and characterized as an adhesive for bonding Ti-6Al-4V was used to bond Celion 6000/LARC-160 composite. Comparisons are made for the two adherend systems. A bonding cycle was determined for the composite bonding and lap shear specimens were prepared which were thermally exposed in a forced-air oven for up to 5000 h at 204 C. The lap shear strengths (LSSs) were determined at RT, 177, and 204 C. After thermal exposure at RT, 177, and 204 C the LSS decreased significantly; however, a slight increase was noted for the 204 C tests. Initially the LSS values are higher for the bonded Ti-6Al-4V than for the bonded composite, however, the LSS decreases dramatically between 5000 and 10,000 h of 204 C thermal exposure. Longer periods of thermal exposure up to 20,000 h results in further decreases in the LSSs. Although the bonded composite retained useful strengths for exposures up to 5000 h, based on the por results of the bonded Ti-6Al-4V beyond 5000 h, the 422 adhesive bonded composites would most likely also produce poor strengths beyond 5000 h exposure. Adhesive bonded composite lap shear specimens exposed to boiling water for 72 h exhibited greatly reduced strengths at all test temperatures. The percent retained after water boil for each test temperature was essentially the same for both systems.

Adhesive Bonding; Polyimides; Shear Strength; Synthesis (Chemistry); Thermoplastic Resins

TOP

#### 19900044453

## Composite developments from fully imidized PMR-15 powder

Hartness, Timothy; Porter, Debra, BASF Structural Materials, Inc., USA; Reardon, Joseph P., Dexter Composites, USA; Jan 1, 1989; 14p; In English; 34th; International SAMPE Symposium and Exhibition, May 8-11, 1989, Reno, NV, USA; See also A90-31501; Copyright; Avail: Issuing Activity

The ability to prepreg a fully imidized PMR-15 that is then consolidated without the evolution of reaction volatiles is a very significant development. PMR-15, the most widely used high-temperature polyimide today, has several severe limitations. The traditional material is supplied as a prepregging varnish. The alcohol varnish contains three monomers that are reacted after prepregging, resulting in the amic acid. One of the monomers, Methylenedianiline, must be handled with care due to its toxic nature. Before curing, the composition of the resin is continually changing because of monomer reactions that occur during storage. Compositional changes of the resin include formation of soluble higher esters, which severely affect the processibility and properties of the composition. These issues are eliminated with the fully imidized product form.

AIAA

Mechanical Properties; Polyimides; Prepregs; Rheology; Thermal Analysis; Thermoplastic Resins

**TOP** 

# 19900046059 Georgia Inst. of Tech., Atlanta, GA, USA

Combining LaRC-TPI powder with carbon fiber by electrostatic fluidized bed coating

Varughese, Babu, Georgia Inst. of Tech., USA; Muzzy, John, Georgia Institute of Technology, USA; Baucom, Robert M., NASA Langley Research Center, USA; Jan 1, 1989; 8p; In English; 21st; International SAMPE Technical Conference, Sept. 25-28, 1989, Atlantic City, NJ, USA; See also A90-33076

Contract(s)/Grant(s): NAS1-864; Copyright; Avail: Issuing Activity

Thermoplastic polyimide prepreg tow is produced rapidly and efficiently by applying the LaRC-TPI matrix as an electrostatically charged and fluidized powder to electrically grounded and spread carbon fiber tow. The powder is melted after coating to insure adhesion to the fibers and to reduce tow friction. Excellent wetout in towpreg samples is obtained resulting in very flexible prepregs. Processing conditions of this towpreg produced with LaRC-TPI powders from Rogers Corp. and Mitsui Toatsu Chemicals are described. Mechanical properties of the towpreg and unidirectional laminates are presented in detail.

Carbon Fibers; Electrostatic Charge; Fluidized Bed Processors; Polyimides; Prepregs; Thermoplastic Resins

**TOP** 

## 19900046075

## Development of a new generation polyamideimide (PAI)

Chen, P. N., Sr.; Vora, R. H.; Glick, M. M.; Jaffe, M. J., Hoechst Celanese Corp., USA; Jan 1, 1989; 12p; In English; 21st; International SAMPE Technical Conference, Sept. 25-28, 1989, Atlantic City, NJ, USA; See also A90-33076; Copyright; Avail: Issuing Activity

This paper provides the synthesis, characterization and development of a new generation of heat stable polyamideimide (PAI) polymers. The polymers are generally prepared by forming the polycondensation product of an aromatic diamine, a trifunctional aromatic anhydride acid chloride and an aromatic dianhydride containing trifluoromethyl moieties. These new materials possess high glass transition temperatures, useful mechanical properties and outstanding thermoplastic flow behavior which render them readily melt processable into fibers, films, sheets and other molded articles. In addition, the as-precipitated fluoro containing PAI materials exhibit solubility in many organic solvents and are thus amenable to solution casting techniques. They also show excellent resistance toward thermooxidative degradation at temperatures to 450 F and low moisture uptake.

AIAA

Amides; Fabrication; Polyimides; Thermal Stability; Thermoplastic Resins

TOP

## 19900063009

# Synthesis of soluble, melt processible polyimides of controlled structure

Waldbauer, R. O.; Rogers, M. E.; York, G. A.; Mcgrath, J. E., NSF, Science and Technology Center; Virginia Polytechnic Institute and State University, USA; Arnold, C. A., ICI Fiberite, Inc., USA; Jan 1, 1990; 11p; In English; 35th; International SAMPE Symposium and Exhibition, Apr. 2-5, 1990, Anaheim, CA, USA; See also A90-50056; Copyright; Avail: Issuing Activity

An effort is made to enhance the processibility of high-T(g) polyimide homopolymers and copolymers suitable for composite matrices and structural adhesives, using solution imidization techniques to convert a variety of poly(amic acid) intermediates to fully cyclized polyimides. Products are thereby obtained which are more soluble than polyimides derived from bulk thermal cyclization of poly(amic acids) at about 300 C. The incorporation of the monofunctional reagent phthalic anhydride is also noted to usefully furnish a control of molecular weight which improves the melt and solution processibility of the resulting polyimides. Tough, transparent films have been prepared on these bases. AIAA

Melting; Polyimides; Polymer Matrix Composites; Thermoplastic Resins

Investigation of the morphological structure and physical behavior of high Tg polyimide thermoplastic homopolymers and siloxane segmented copolymers

York, G. A.; Waldbauer, R. O.; Rogers, M. E.; Gungor, A., NSF, Science and Technology Center; Virginia Polytechnic Institute and State University, USA; Arnold, C. A., ICI Fiberite, Inc., USA; Jan 1, 1990; 11p; In English; 35th; International SAMPE Symposium and Exhibition, Apr. 2-5, 1990, Anaheim, CA, USA; See also A90-50056; Copyright; Avail: Issuing Activity

To determine the mechanical properties and morphological structure of siloxane polyimide segmented copolymers which find application in aerospace and microelectronics, a study utilizing transmission electron microscopy (TEM), thermal analyses, and mechanical analysis has been conducted. The TEM analysis demonstrated the presence of extremely fine (less than 10 nm) spherical siloxane microdomains in the bulk for both 2000 and 10,000 Mn segment size siloxane systems. The thermal analysis showed that the degree of mixing between the polydimethilsiloxane (PDMS) segments and the polyimide segments was strongly dependent on the PDMS molecular weight, the chemical nature of the polyimide segment, and the processing conditions. The shifting of the system to a less polar one resulted in a phase shift from semicontinuous to discrete spherical morphology and an increase of the modulus.

Copolymers; Morphology; Polyimides; Polymerization; Siloxanes; Thermal Analysis; Thermoplastic Resins

**TOP** 

## 19900063079 Mitsui Toatsu Chemicals, Inc., Yokohama, Japan

Improved melt flow and physical properties of Mitsui Toatsu's LARC-TPI 1500 series polyimide

Ohta, Masahiro, Mitsui Toatsu Chemicals, Japan; Tamai, Shoji, Mitsui Toatsu Chemicals, Inc., Japan; Towell, T. W., Mitsui Toatsu Chemicals, Japan; Johnston, N. J., Mitsui Toatsu Chemicals, Japan; Saint Clair, T. L., NASA Langley Research Center, USA; Jan 1, 1990; 15p; In English; 35th; International SAMPE Symposium and Exhibition, Apr. 2-5, 1990, Anaheim, CA, USA; See also A90-50056; Copyright; Avail: Issuing Activity

The basic thermal properties and melt behavior of an improved form of LARC-TPI polyimide, LARC-TPI 1500, are investigated and compared with those of LARC-TPI 1000 and 2000. The LARC-TPI 1500 powders are thermally stable and melt processable. The absolute molecular weight of the polymer varies from 20,000 to 30,000, indicating recurring unit numbers of about 40-60. The melt flow properties of LARC-TPI 1500 are comparable to those of other engineering plastics, such as PEEK, PSU, and PES. The flexural modulus of injection molded parts of LARC-TPI 1500 is the highest among commercial thermoplastic polymers.

Melting; Physical Properties; Polyimides; Resin Matrix Composites; Thermoplastic Resins

**TOP** 

#### 19900063144

Effect of molecular weight and end group control on the adhesion behavior of thermoplastic polyimides and poly(imide siloxane) segmented copolymers

Yoon, T. H.; Mcgrath, J. E., NSF; Virginia Polytechnic Institute and State University, USA; Arnold, C. A., ICI Fiberite, Inc., USA; Jan 1, 1990; 13p; In English; 35th; International SAMPE Symposium and Exhibition, Apr. 2-5, 1990, Anaheim, CA, USA; See also A90-50056; Copyright; Avail: Issuing

Adhesives; Copolymers; Molecular Weight; Polyimide Resins; Polyimides; Thermoplastic Resins

**TOP** 

# 19910064466 Lockheed Engineering and Sciences Co., Hampton, VA, USA

Film properties of high performance semi-interpenetrating polyimide networks
Gerber, M. K., Lockheed Engineering and Sciences Co., USA; Pater, R. H., NASA Langley Research Center, USA; Jan 1, 1990; 3p; In English; 48th; SPE Annual Technical Conference, May 7-11, 1990, Dallas, TX, USA; See also A91-49076; Copyright; Avail: Issuing Activity

Consideration is given to an approach to provide a semi-IPN in which two constituent materials have nearly the same chemical structure and polymer chain length. A reactive endcapped LARC-TPI polyamic acid having a formulated molecular weight of 20,000 is prepared and then combined with various quantities of a commercial LARC-TPI polyamic acid solution. The polyimide films of semi-IPNs are tested along with two constituent materials. The physical and mechanical properties of the polyimide films are presented. The applicability of using this approach to minimize phase separation and to maximize long-term phase stability is discussed. The concept of controlling morphology by chemistry is proven for providing LARC-TPI based semi-2-IPNs. A very long chain LARC-TPI endcapped with a nadic group exhibits physical properties almost identical to unendcapped LARC-TPI.

Polyimides; Thermoplastic Films

**TOP** 

# 19930028750 NASA Langley Research Center, Hampton, VA, USA

Melt flow properties of LARC-TPI 1500 series mixtures

Burks, H. D., NASA Langley Research Center, USA; St. Clair, T. L., NASA Langley Research Center, USA; SAMPE Quarterly; Oct. 1992; ISSN

0036-0821; 24, 1, pp. 50-53.; In English; Copyright; Avail: Issuing Activity

This study examines the melt-flow properties of 50/50 mixtures of high flow grade/medium flow grade LARC-TPI 1500 series pellets and a new form (94/6), of medium flow grade material which contains 6 percent of a low molecular weight imide additive. The flow properties are compared with previously reported flow properties of mixtures of earlier batches of high and medium flow grade TPI 1500 materials. Unlike the earlier batches of materials, the later materials were synthesized and mixed at Mitsui Toatsu Chemicals, Inc., Japan, and made into pellets before NASA received them for characterization. They exhibited a lower melt viscosity as well as improved melt stability. AIAA

Aromatic Compounds; Melting; Molecular Weight; Polyimides; Thermoplastic Resins

**TOP** 

# 19930068770 NASA Langley Research Center, Hampton, VA, USA

A thermoplastic copolyimide

Progar, D. J., NASA Langley Research Center, USA; St. Clair, T. L., NASA Langley Research Center, USA; Oct 1, 1985, pp. 31 p.; In English; 2nd; Society of Plastics Engineers, International Conference on Polyimides, Oct. 30, 1985-Nov. 1, 1985, Ellenville, NY, USA; Sponsored by Society of Plastics Engineers; Avail: Issuing Activity

A copolyimide, STPI/LARC, was prepared from the reaction of 3,3'4,4'-benzophenonetetracarboxylic dianhydride (BTDA), equimolar quantities of m-phenylenediamine and 4,4'-oxydianiline, and a small amount of phthalic anhydride to control the molecular weight. The processability and adhesive properties of STPI/LARC were compared to those of a commercially available form of LARC-TPI. LARC-TPI, a thermoplastic polyimide, from the reaction of BTDA and 3,3'-diaminobenzophenone, had previously shown promise as a high temperature structural adhesive. Lap shear specimens were fabricated using adhesive tape prepared from each of the two polymers. Lap shear tests were performed at room temperature, 177 C, and 204 C before and after exposure to a 72-hour water-boil and to aging at 204 C. Adhesive Bonding; Fabrication; Mechanical Properties; Polyimides; Thermoplastic Resins

A new thermoplastic polyimide composite prepared by the polymerization of monomer reactants approach

Zhou, Jiang; He, Tianbai; Zhang, Jin; Ding, Mengxian, Chinese Academy of Sciences, China; SAMPE Quarterly; July 1993; ISSN 0036-0821; 24, 4, pp. 31-34.; In English; Copyright; Avail: Issuing Activity

A novel amorphous thermoplastic polyimide (PTI) is being developed as a potential matrix resin for advanced composites. This paper describes the manufacture of the resin, prepreg, and processing of the composite. The chemical and physical behavior of the resin during the processing was determined by infrared spectroscopy and rheology. The influence of processing conditions on the composite properties was investigated. Mechanical properties of the unidirectional carbon fiber/PTI laminates were also presented.

Carbon Fibers; Fiber Composites; Laminates; Polyimides; Polymer Matrix Composites; Thermoplastic Resins

**TOP** 

## 19950007696 NASA Langley Research Center, Hampton, VA, USA

Copolyimides prepared from ODPA, BTDA and 3,4'-ODA

Chang, Alice C., inventor, Lockheed Engineering and Sciences Co., USA; St.clair, Terry L., inventor, NASA Langley Research Center, USA; Aug 31, 1994; 15p; In English

Patent Info.: NASA-CASE-LAR-15109-1; US-PATENT-APPL-SN-299172

Report No.(s): NAS 1.71:LAR-15109-1; Avail: CASI; A03, Hardcopy; A01, Microfiche

A copolyimide was prepared by reacting 3,4'-oxydianiline (3,4'-ODA) with a dianhydride blend comprising, based on the total amount of the dianhydride blend, about 67 to 80 mole percent of 4,4'-oxydiphthalic anhydride (ODPA) and about 20 to 33 mole percent of 3,3',4,4'-benzophenonetetracarboxylic dianhydride (BTDA). The copolyimide may be endcapped with up to about 10 mole percent of a monofunctional aromatic anhydride and has unbalanced stoichiometry such that a molar deficit in the dianhydride blend is compensated with twice the molar amount of the monofunctional aromatic anhydride. The copolyimide was used to prepare composites, films and adhesives. The film and adhesive properties were significantly better than those of LaRC(TM)-IA. NASA

Adhesion; Adhesive Bonding; Anhydrides; Polyimides; Stoichiometry; Thermoplastic Resins

**TOP** 

# 19950009681 Virginia Polytechnic Inst. and State Univ., Blacksburg, VA, USA

Thermoplastic and thermosetting high glass transition temperature matrix resins Final Report, 1 May 1991 - 30 Jun. 1993

Mcgrath, James E., Virginia Polytechnic Inst. and State Univ., USA; May 1, 1994; 4p; In English

Contract(s)/Grant(s): DAAL03-91-G-0140

Report No. (s): AD-À281508; Avail: Issuing Activity (Defense Technical Information Center (DTIC)), Unavail. Microfiche; Limited Reproducibility: More than 20% of this document may be affected by microfiche quality

High performance polymers based upon 3-F containing diamines and bisaminophenols were prepared via careful molecular design. Extremely high glass transition temperatures of more than 430 C could be produced while still maintaining solubility in organic solvents. Alternatively, phenyl ethynyl endcaps were synthesized, which permitted solvent resistant cured composite matrix resins and adhesives to be prepared. With proper design, crystallizable semicrystalline materials, which melted over 400 C, were also prepared. Selected copolyimides were prepared that appear to be melt-processable and still show melting characteristics greater than 400 C. Knowledge transfer of these findings was successfully achieved in cooperation with the Lockheed Company and, as a result, void-free panels were prepared showing cured glass transition temperatures of 700 F or higher.

Glass Transition Temperature; High Temperature; Matrix Materials; Polyimide Resins; Polyimides; Thermal Stability; Thermoplastic Resins; Thermosetting Resins

**TOP** 

# 19960000927 Adhesion Society, Inc., Blacksburg, VA, USA

Proceedings of the 18th Annual Meeting of the Adhesion Society Final Report, 15 Nov. - 30 Sep. 1995

Feb 22, 1995; 398p; In English, 19-22 Feb. 1995, Hilton Head, SC, USA

Contract(s)/Grant(s): N00014-95-1-0216 Report No.(s): AD-A294748; Avail: CASI; A17, Hardcopy; A04, Microfiche

This document is a collection of abstracts of papers presented at the 18th Annual Adhesion Society Meeting held at Hilton Head, SC on February 19-22, 1995. The program was chaired by Joseph W. Holubka. Three areas of adhesion science will be emphasized in this years meeting. These areas include: New Materials for Adhesives and Coatings; Small Particle Adhesion; and Chemical/Physical Characterization of Adhesive Interactions. Two sessions will be assigned to each of these areas of technical interest. In addition, 'special topics sessions' on modelling adhesion processes; durability: environmentally induced adhesion reduction, pressure sensitive adhesives; mechanical characterization adhesion producing interphases; and applications of adhesion science will also be presented.

Acrylic Resins; Adhesion; Adhesive Bonding; Adhesives; Ceramics; Characterization; Composite Materials; Conferences; Phenolic Epoxy Resins; Plastics; Polyimides; Radioactive Materials; Reinforcing Fibers; Stainless Steels; Thermoplastic Resins

**TOP** 

# 19960051555

# Effect of crystallization of thermoplastic polyimide resin on erosion rate

Miyazaki, N., Kyushu Univ, Japan; Journal of Materials Science Letters; August 15 1996; ISSN 0261-8028; 15, 16, pp. 1422-1423; In English; Copyright; Avail: Issuing Activity

As a study of erosion behavior of plastic materials, sand erosion tests of a thermoplastic polyimide resin were performed. An amorphous resin and crystalline resins, whose degrees of crystallinity were 15, 25 and 30%, were prepared to study the effects of resin crystallization on the erosion rate. A sand-blast type of test apparatus was used in the erosion tests. It is concluded that the erosion rate increases with increasing crystallinity, because plastic materials become hard with increasing crystallinity Author (EĬ)

Cavitation Corrosion; Crystallization; Hardness; Polyimide Resins; Polyimides; Thermoplastic Resins

**TOP** 

## 19970036643

# Blends of thermoplastic polyimide with epoxy resin

Kimoto, Masaki, Technology Research Inst of Osaka Prefecture, Japan; Mizutani, Kiyoshi; Journal of Materials Science; May 1 1997; ISSN 0022-2461; 32, 9, pp. 2479-2483; In English; Copyright; Avail: Issuing Activity

Bifunctional epoxy resins have been modified with thermoplastic polyimide in order to improve their toughness. The effects of polyimide content, curing conditions, curing agents, and types of polyimide on the fracture properties have been examined. Relationships between the microstructure of the cured resins and their mechanical properties have also been investigated.

Epoxy Resins; Polyimides; Polymer Blends; Thermoplastic Resins

## Imide oligomer modified polyimides for improved composite processing

Vaccaro, Eleonora, Connecticut, Univ., Storrs, USA; Scola, Daniel A., Connecticut, Univ., Storrs; 1996, pp. 39-47; In English; Copyright; Avail:

The high melt viscosities of high temperature polyimide resins require high pressures (1000 psi) for consolidation to produce low void composites. It would be desirable to have a low melt, low viscosity polyimide system which can be processed at lower pressure (100 to 300 psi) to produce low void composites in high yield. A series of low molecular weight, low viscosity imide oligomers has been synthesized and blended with polyimides. Graphite fiber composites were fabricated from the oligomer-modified polyimide resins/graphite fiber prepreg. The quality of the composites is discussed in terms of the resin flow and void contents.

Author (AIAA)

Oligomers; Polyimides; Viscosity; Graphite-Polyimide Composites; Thermoplastic Resins

**TOP** 

#### 19980079908

## Thermal characterization of new high performance semi-interpenetrating polymer networks

Pater, R. H., NASA Langley Research Center, USA; Ray, Asit K., Christian Brothers Univ., USA; 1996, pp. 198-206; In English; Copyright; Avail: AIAA Dispatch

Semi-interpenetrating polymer networks (semi-IPNs) of thermoset LARC-IRP 46 and thermoplastic LARC-IA polyimides were prepared in weight percent ratios ranging from 100:0 to 0:100. Thermal analysis and dynamic mechanical analysis were made on these samples to correlate their chemical composition with physical/mechanical/morphological properties. As expected, the glass transition temperature increases with an increase in thermoset concentrations. The opposite is true for the thermo-oxidative stability. All the semi-IPNs showed unusually good phase-mixing. The resin density goes through a maximum at about 50:50 weight ratio, suggesting that the semi-IPN samples are not merely a solid solution of the two polymeric components. This nonlinear behavior may be interpreted in terms of the existence of an electrostatic interaction between the two constituent polymers. Author (AIAA)

Thermosetting Resins; Polyimides; Thermoplastic Resins; Penetration; Polymerization

**TOP** 

#### 19980087372

# Preparation and stress relaxation properties of thermoplastic polysiloxane-block-polyimides

Furukawa, Nobuyuki, Nippon Steel Chemical Co., Ltd., Japan; Yamada, Yasuharu, Nippon Steel Chemical Co., Ltd., Japan; Kimura, Yoshiharu, Kyoto Inst. of Technology, Japan; High Performance Polymers; Dec. 1996; ISSN 0954-0083; Volume 8,, no. 4, pp. 617-630; In English; Copyright; Avail: AIAA Dispatch

Three series of thermoplastic polysiloxane-block-polyimides were prepared from 3,3',4,4'-benzophenonetetracarboxylic dianhydride, aromatic diamines with four phenylene rings, and diamine-terminated polydimethylsiloxane (Mw = 1240). The tensile modulus of their films was lowered, and the coefficient of thermal expansion was increased with increasing polysiloxane composition. The copolyimides showed a good adherence onto silicon wafer even without adhesion promoter added. The adherence was maintained even after exposure to an environment of 23 C and 78 percent RH for 72 h. The residual stress in the interface between the copolyimide coating and the silicon wafer was lowered according to the tensile modulus of coating materials, so the introduction of polysiloxane into aromatic polyimides can afford excellent adhesion and stress relaxation properties despite mismatching in thermal expansion between polymer and substrate.

Author (AIAA)

Stress Relaxation; Thermoplastic Resins; Polysiloxanes; Polyimides; Mechanical Properties; Thermophysical Properties

**TOP** 

## 19980118079

# Elastic modulus of the crystalline regions of thermoplastic polyimide

Nakamae, Katsuhiko, Kobe Univ., Japan; Nishino, Takashi, Kobe Univ., Japan; Miki, Norihiko, Kobe Univ., Japan; High Performance Polymers; Sep. 1995; ISSN 0954-0083; Volume 7,, no. 3, pp. 371-376; In English; Copyright; Avail: Aeroplus Dispatch

The elastic modulus E1 of the crystalline regions in the direction parallel to the chain axis was measured for poly/3,3',4,4'-(dioxy biphenyl) diphenylene pyromellite imide/ (New-TPI) by X-ray diffraction. An E1 value of 55 GPa was obtained for New-TPI. This small E1 value was considered to be due to the coexistence of both ether bonds and m-phenylene linkages which are introduced to improve the processability. The elastic moduli Et of the crystalline regions in the direction perpendicular to the chain axis were 3.2-3.5 GPa. These small Et values indicate that any special interaction hardly acts for New-TPI.

Author (AIAA)

Modulus of Elasticity; Thermoplastic Resins; Polyimides; X Ray Diffraction; Crystallinity

TOP

## 19980169132

# Polyimides based on 2,2',6,6'-tetramethylbenzidine

Chuang, Kathy C., NASA Lewis Research Center, USA; High Performance Polymers; Mar. 1995; ISSN 0954-0083; Volume 7,, no. 1, pp. 81-92; In English; Copyright; Avail: Aeroplus Dispatch

Thermoplastic and addition-curing polyimides based on non-coplanar 2,2',6,6'-tetramethylbenzidine (TMBZ) were prepared and investigated. The TMBZ-based thermoplastic polyimides displayed high Tg values, enhanced solubility, and excellent thermal stability. These high-molecular-weight polyimides could be either cast into optically transparent thin films or spun into fibers. The addition-curing polyimides based on TMBZ exhibited higher Tg values than the corresponding PMR-II type polyimides, due to the increased rotational barrier created by the substituents on the biphenyl moiety. However, the methyl substituents on the TMBZ inherently lowered the thermo-oxidative stability of TMBZ-based polyimides, relative to the corresponding PMR-II type polyimides. Author (AIAA)

Polyimides; Curing; Thermoplastic Resins; Physical Properties; Polymerization

Miscibility, morphology and tensile properties of the melt blends of liquid crystalline polyimide (PI-LC) with two different thermoplastic polyimides - Semicrystalline polyimide (N-TPI) and amorphous poly(ether-imide) (PEI)

Konda, M., Kyoto Inst. of Technology, Japan; Tanaka, M., Kyoto Inst. of Technology, Japan; Miyamoto, M., Kyoto Inst. of Technology, Japan; Kimura, Y., Kyoto Inst. of Technology, Japan; Yamaguchi, A., Mitsui Toatsu Chemicals, Inc., Japan; High Performance Polymers; Mar. 1998; ISSN 0954-0083; Volume 10, no. 1, pp. 93-109; In English; Copyright; Avail: Aeroplus Dispatch

A liquid crystalline polyimide (poly (1,3-bis (4-(4-aminophenoxy) - alpha,alpha - dimethylbenzyl) benzene pyromellitimide): PI-LC) was blended with two kinds of thermoplastic polyimide: poly (3,3'-(4,4'-dioxybiphenyl) diphenylene pyromellitimide) (N-TPI), which is a semicrystalline polyimide having a melting point of 388 C and poly (2,2'-bis (3,4-dicarboxyphenoxy) phenylpropane-2-phenylene bisimide) (PEI), which is an amorphous poly(etherimide) having a glass transition temperature at 220 C. The miscibility of N-TPI and PI-LC was evaluated by DSC using enthalpy relaxation, and the phase diagram of their blend was drawn above the melting point of N-TPI. The result indicated that the PI-LC-rich blend was miscible in a wide range of compositions. However, the extruded samples of their blend, which had been prepared under a flow of high shear, showed a phase separation structure. In the other combination of PEI and PI-LC, the blend was immiscible, but at low composition of PI-LC their blend fiber showed higher tensile properties than the original PEI fiber.

Solubility; Morphology; Tensile Properties; Liquid Crystals; Polyimides; Thermoplastic Resins

**TOP** 

19990078759 Clark-Atlanta Univ., GA USA

Computational Materials Research, 15 Dec. 1997 - 14 Dec. 1998

Veazie, David R., Clark-Atlanta Univ., USA; 1998; 16p; In English

Contract(s)/Grant(s): NCC1-270; No Copyright; Avail: CASI; AO3, Hardcopy; AO1, Microfiche

High temperature thermoplastic polyimide polymers are incorporated in engineering structures in the form of matrix materials in advanced fiber composites and adhesives in bonded joints. Developing analytical tools to predict long term performance and screen for final materials selection for polymers is the impetus for intensive studies at NASA and major industry based airframe developers. These fiber-reinforced polymeric composites (FRPCs) combine high strength with lightweight. In addition, they offer corrosion and fatigue resistance, a reduction in parts count, and new possibilities for control through aeroelastic tailoring and "smart" structures containing fully-integrated sensors and actuators. However, large-scale acceptance and use of polymer composites has historically been extremely slow. Reasons for this include a lack of familiarity of designers with the materials; the need for new tooling and new inspection and repair infrastructures; and high raw materials and fabrication costs.

Author

High Temperature; Thermoplasticity; Thermoplastic Resins; Polyimides; Polyimide Resins

**TOP** 

#### 19990090307

High solid precursor solution of thermoplastic polyimides

Echigo, Yoshiaki, Unitika Ltd., Japan; Kaneshiro, Hisayasu; Journal of Polymer Science, Part A: Polymer Chemistry; Jan 01, 1999; ISSN 0887-624X; Volume 37, no. 1, pp. 11-14; In English; Copyright; Avail: Issuing Activity

A preparative method of high solid thermoplastic polyimide (TPI) precursor solutions is investigated. Results showed that an equimolar mixture of tetracarboxylic acids and diamines can be used as precursors for LaRC IA, IAX, or 8515. These precursor high solid solutions can afford transparent, flexible TPI films by a casting method involving subsequent thermal cure. The aspects of these TPI precursor solutions are presented.

Polyimides; Thermoplastic Resins; Solid Solutions; Carboxylic Acids

**TOP** 

## 19990103757

Semicrystalline thermoplastic polyimide + polymer liquid crystal blends: Nonisothermal calorimetry and thermogravimetry

Brostow, Witold, Univ. of North Texas, USA; D'Souza, Nandika Anne; Gopalanarayanan, Bhaskar; Polymer Engineering and Science; Jan, 1998; ISSN 0032-3888; Volume 38, no. 1, pp. 204-212; In English; Copyright; Avail: Issuing Activity

Blends of a thermoplastic polyimide (TPI) and a polymer liquid crystal (PLC) were studied using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The presence of PLC enhances orientation in the system and lowers the crystallization temperature - a manifestation of the channeling effect predicted theoretically (38). The degradation studies show high temperature stability of all blends with the degradation onset consistently above 520 C. The onset decreases with PLC concentration but reaches a plateau above 30 wt% PLC when the PLC-rich islands are formed. The amount of moisture absorbed decreases with the PLC concentration while the moisture does not affect the degradation of the samples significantly. A phase diagram is constructed for the PLC + TPI blends from the DSC and TGA data. A comparison of amorphous and semicrystalline TPI and the characterization of amorphous TPI + PLC blends will be reported later.

Author (EI)

Heat Measurement; Polymer Blends; Liquid Crystals; Polymerization; Polymides; Thermoplastic Resins

**TOP** 

# Mechanical Engineering

Includes mechanical devices and equipment; machine elements and processes. For cases where the application of a device or the host vehicle is emphasized see also the specific category where the application or vehicle is treated.

## 19930069432

Structural applications of Avimid K3B LDF thermoplastic composites

Perrella, Andrew P., Du Pont Composites, USA; In: International SAMPE Technical Conference, 24th and International SAMPE Metals and Metals Processing Conference, 3rd, Toronto, Canada, Oct. 20-22, 1992, Proceedings. Vol. 24 (A93-53376 23-23); 1992, pp. T711-T723.; In English; Copyright; Avail: Issuing Activity

Composite applications on advanced aircraft require lightweight, high performance, tough material systems which are capable of operating at high service temperatures. These composite systems must also be producible and cost effective. Avimid K3B composite materials and related process and part manufacturing technologies offers a unique solutions to these requirements. The objective of this paper is to describe selected Avimid K3B processing approaches such as Long Discontinuous Fiber thermoforming and fusion bonding. A review of the Avimid K3B F-16 Strake Door Joint Development Program is presented. This program successfully developed, built and structurally validated a flight demonstration component using these materials and manufacturing methods.

Aircraft Structures; Composite Structures; Forming Techniques; Fusion Welding; Polyimides; Thermoplastic Resins

# **Solid-State Physics**

Includes condensed matter physics, crystallography, and superconductivity.

19930003834 North Carolina State Univ., Coll. of Textiles., Raleigh, NC, USA

The crystallization and crystalline properties of LARC-TPI Final Technical Report, 1 Jan. 1987 - 31 Dec. 1988

Theil, Michael H., North Carolina State Univ., USA; Gangal, Pravin D., North Carolina State Univ., USA; Jan 1, 1992; 36p; In English Contract(s)/Grant(s): NAG1-723

Report No.(s): NASA-CR-191001; NAS 1.26:191001; Avail: CASI; A03, Hardcopy; A01, Microfiche

LARC-TPI, a thermoplastic polyimide, has been studied in order to develop an understanding of its crystalline phase transition. Our experiments suggest that samples synthesized in different laboratories apparently had different degrees of imidization and their thermal behaviors differed accordingly. When the most crystalline of these polyimides was studied in some detail, we found that it melted irreversibly in that once a sample was completely melted it would not recrystallize. A polymer that did not recrystallize displayed a glass transition, which increased in temperature upon subsequent cooling and reheating. Solubility experiments indicated that heating above the crystalline melting temperature led to network formation in the polymer, a conclusion that is consistent with other behavior just mentioned. Differential calorimetric studies revealed that annealing at slow heating rates or under isothermal conditions resulted in dual melting transitions. These studies, supported by X-ray diffraction results, strongly indicate that the annealing process involves a solid-liquid-solid transformation. From an existing phenomen- ological model for the kinetics of phase transitions, kinetic parameters for these crystallizations have been evaluated. The Avrami exponents n increased with the annealing temperature in the protocol used in this study. Their values were about 2 or lower, thus indicating that crystallization may have followed a mechanism that included heterogeneous nucleation of a low dimensional order in which all the embryonic crystallites formed at the beginning of the process. A positive temperature coefficient for these crystallizations indicated that diffusion may have had a rate controlling influence and affected the values of n. Author

Annealing; Crystallinity; Phase Transformations; Polyimides; Recrystallization; Thermoplastic Resins